This Page Is Inserted by IFW Operations and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

As rescanning documents will not correct images, please do not report the images to the Image Problem Mailbox.

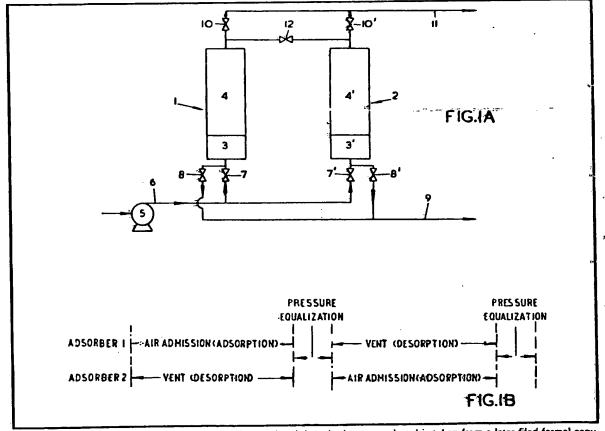
(12) UK Patent Application (19) GB (11) 2 042 365 A

- (21) Application No 7907721
- (22) Date of filing 5 Mar 1979
- (43) Application published 24 Sep 1980
- (51) INT CL3 BO1D 53/04
- (52) Domestic classification B1L 101 102 201 209 211 215 217 223 302 306 307 402 BC
- (56) Documents cited GB 1460595 GB 1449864 GB 1012349
- (58) Field of search B1L
- (71) Applicant
 BOC Limited
 Hammersmith House
 London W6 9DX
- (72) Inventor
 Kenneth Cecil Smith
- (74) Agents R W Obee

(54) Gas separation

(57) A pressure swing adsorption (PSA) process for the separation of a component from a gas mixture also containing water vapour, eg the separation of nitrogen from air, employs adsorbers 1 and 2 each having a first stage 3, 3' containing a desiccant adsorbent and a second stage 4, 4' containing an adsorbent which preferentially adsorbs another component of the mixture eg a carbon molecular sieve which adsorbs oxygen more rapidly than nitrogen. Each adsorber is put through a cycle comprising an adsorption step during which the mixture is passed through the adsorber by a compressor 5 and unadsorbed product is withdrawn through a delivery line 11, and a desorption step during which the adsorber is vented to atmosphere through a waste gas

line 9. The pressure reached in the adsorber during such venting is the lowest reached during the entire cycle, no vacuum pump being employed. A valved line 12 allows pressure equalisation between the adsorbers.



The drawings originally filed were informal and the print here reproduced is taken from a later filed formal copy.

GB 2 042 365 A

SPECIFICATION

Gas separation

5 The present invention relates to a method of gas separation by pressure swing adsorption (PSA) techniques, and is particularly, though not exclusively, concerned with a PSA process for the separation of nitrogen from air.

One known PSA process for the separation of nitrogen from air employs a molecular sieve carbon adsorbent which has the ability to effect a separation as between the two major components of air by virtue of its more rapid adsorption of oxygen than of nitrogen. In operation a bed of this adsorbent is put through a cycle which includes an adsorption step during which time air is pumped through the bed, most of the oxygen and a proportion of the nitrogen plus substantially all of the carbon dioxide and water vapour in the feedstock are adsorbed, and a nitrogen rich product gas is supplied from the outlet of the bed; and

a desorption step during which time the outlet

25 of the bed is closed, the bed is vented to
atmospheric pressure through its inlet and
then evacuated through its inlet, so that the
adsorbed gases are substantially removed
from the bed thereby preparing it for the next

30 adsorption step. In practice to adsorbent beds

adsorption step. In practice to adsorbent beds are employed and operated on similar cycles but sequenced to be out of phase with one another by 180°, so that when one bed is on its adsorption step the other bed is on its

35 desorption step, and vice versa. Furthermore it is usual to equalise the pressures in the two beds between each step by connecting the two bed inlets together and connecting the two bed outlets together; with these connections and the statement of the statem

40 tions made the gas within the void spaces of the bed which has just completed its adsorption step is sucked into the bed which has just completed its desorption step by virtue of the pressure difference which exists between the

45 beds at that stage, and this is found to be beneficial in maximising the product output because such void space gas will have already become somewhat enriched in nitrogen.

To effect the feed of air through the adsor50 bent beds a plant operating this process must include a compressor, and to evacuate the beds during desorption a vacuum pump is also included. Typically the bed pressure during adsorption rises sto a maximum value of 7 to 10 bar g (although lower adsorption pressures are sometimes employed), with the vacuum reached during desorption typically being

in the region of 100 Torr. Plants operating the process as described above can readily 60 achieve a nitrogen product purity in excess of 99% by volume.

Now, in order to reduce the capital and running costs of such a plant it has been proposed to eliminate the vacuum pump, so that during desorption the beds are vented to

atmospheric pressure but are not reduced to a pressure below atmospheric. Clearly if costs are reduced in this manner a penalty will be paid in that, for the same product purity and

70 all other factors being equal, the plant will not be able to process the same volume of feedstock as a plant operating with a vacuum pump and thereby providing a greater overall pressure swing. Nevertheless, if the after-men-

75 tioned problem can be overcome such operation may make economic sense in many instances, and the operator of a plant initially assembled without a vacuum pump is given the flexibility to up-rate the performance of his

80 plant at a later date if circumstances should so require, simply by installing a vacuum pump.

The problem which has been found in oper-

ating the process without a vacuum pump is that, over a period, a gradual decrease in the 85 product output at a given purity is observed, in one experiment the product flow rate falling by some 40% over a period of six hundred hours. From a consideration of the water adsorption isotherm of the molecular sieve

90 carbon adsorbent we postulate that the reason for this decreasing performance is that, without vacuum, a proportion of the water vapour adsorbed during the adsorption step remains on the adsorbent after the desorption step, so

95 that as the cycle is repeated moisture gradually builds up in the adsorbent bed thereby decreasing its capacity to effect the desired oxygen/nitrogen separation.

It is an aim of the invention to overcome 100 this problem and, while the invention is principally concerned with the separation of nitrogen from air using a molecular sieve carbon adsorbent the invention may also be applicable to other PSA separations where similar

105 problems arise. As an example there can be quoted the separation of hydrogen from a mixture with methane using the same carbon adsorbent and where moisture is also present in the feedstock.

110 In a broad aspect the invention accordingly resides in a process for the separation of a desired gaseous component from a gaseous mixture including the desired component, at least one other component and water vapour,

115 which process comprises repeating a cycle of operation including successive adsorption and desorption steps, the adsorption step comprising passing the gaseous mixture at a superatmospheric pressure through an adsorber hav-

120 ing a first stage containing a desiccant adsorbent and a second stage containing an adsorbent which adsorbs said other component more readily than said desired component, so as to obtain from the adsorber a product gas

125 enriched in said desired component and depleted in said other component and water vapour; and the desorption step comprising venting the adsorber to a pressure not substantially below atmospheric pressure in a

130 direction counter to the direction of passing

to effect a nitrogen/oxygen separation in the same way as the beds of a plant operating the known process previously described, but that no vacuum pump is included in the plant thereby reducing both its capital and running costs. Furthermore, the incorporation of desic-

costs. Furthermore, the incorporation of desiccant beds 3 and 3' which effectively function as heatless driers upstream of the main carbon beds ensures that there can be no unde-10 sirable build up of moisture in the adsorbers.

Turning now to Fig. 2A this shows a modification of the Fig. 1A plant to provide an additional purging operation during the desorption steps of each adsorber. To this end a purging line 13 including a throttle valve 13A branches from the product delivery line 11 and in turn branches into two lines leading to the outlet ends of the adsorbers 1 and 2 controlled respectively by valves 14 and 14'.

20 At a predetermined interval after the opening, of the valve 8 or 8' during the desorption step of the appropriate adsorber the valve 14 or 14' is also opened and remains open with valve 8 or 8' until the end of the desorption

25 step. During this time a proportion of the product gas from line 11 is diverted through line 13 (the proportion being determined by the setting of throttle valve 13A) and flows downwardly through the appropriate adsorber
20 the set of the s

30 thereby purging out the void space gas from each of the adsorber stages and replacing such gas with product-quality gas in preparation for the following adsorption step. Such a purging step may be especially valuable in an

35 embodiment having the same adsorbents as described above but used for separating hydrogen from a mixture with methane and water vapour.

As a further variation of either plant indi-40 cated in broken line in Fig. 2A an additional line controlled by a valve 15 may be provided whereby the inlet ends of the adsorbers can

be connected together during the pressure

equalization steps.

CLAIMS

1. A process for the separation of a desired gaseous component from a gaseous mixture including the desired component, at least 50 one other component and water vapour, which process comprises repeating a cycle of operation including successive adsorption and desorption steps, the adsorption step comprising passing the gaseous mixture at a superat-55 mospheric pressure through an adsorber having a first stage containing a desiccant adsorbent and a second stage containing an adsorbent which adsorbs said other component more readily than said desired component, so 60 as to obtain from the adsorber a product gas enriched in said desired component and depleted in said other component and water vapour; and the desorption step comprising venting the adsorber to a pressure not sub-65 stantially below atmospheric pressure in a

direction counter to the direction of passing the gaseous mixture, such pressure being the minimum pressure obtained in the adsorber during the cycle.

70 2. A process for the separation of nitrogen from a gaseous mixture including nitrogen, oxygen and water vapour, which process comprises repeating a cycle of operation including successive adsorption and desorption steps,

75 the adsorption step comprising passing the gaseous mixture at a superatmospheric pressure through an adsorber having a first stage containing a desiccant adsorbent and a second stage containing a molecular sieve carbon

80 adsorbent which adsorbs oxygen more rapidly than nitrogen, so as to obtain from the adsorber a product gas enriched in nitrogen and depleted in oxygen and water vapour; and the desorption step comprising venting the adsor-

*85 ber to a pressure not substantially below atmospheric pressure in a direction counter to the direction of passing the gaseous mixture, such pressure being the minimum pressure obtained in the adsorber during the cycle.

90 3. A process according to claim 1 or claim 2 wherein said desiccant adsorbent is alumina or silica gel.

4. A process according to any preceding claim wherein the desorption step includes,

95 subsequent to venting the adsorber, purging the adsorber with a stream of said product gas in a direction counter to the direction of passing the gaseous mixture.

5. A process according to any preceding 100 claim wherein the maximum pressure reached within the adsorber as a result of the adsorption step is not above 10 bar g

A process according to any preceding claim employing two such adsorbers and

105 wherein each undergoes a similar such cycle of operation but sequenced to be out of phase with one another by 180° so that when one adsorber is on its adsorption step the other adsorber is on its desorption step, and vice 110 versa.

 A process according to claim 6 which includes the step of equalizing the pressures within the two adsorbers between each adsorption and desorption step.

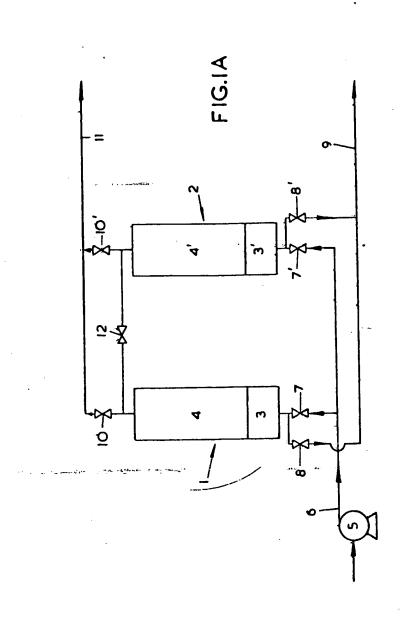
15 8. A process according to claim 7 wherein said pressure equalization is achieved by connecting together the outlets of the adsorbers.

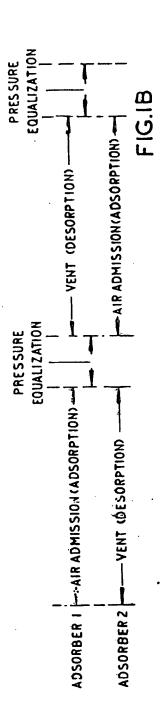
9. A process according to claim 7 wherein said pressure equalization is achieved by con-

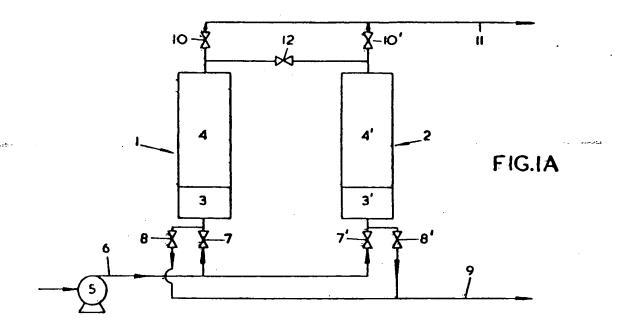
120 necting together the outlets of the adsorbers and connecting together the inlets of the adsorbers.

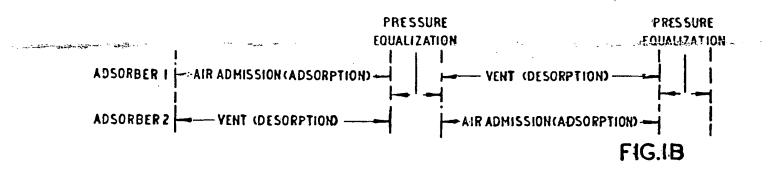
10. A process for the separation of a desired gaseous component from a gaseous
125 mixture including the desired component, at least one other component and water vapour, substantially as hereinbefore described with reference to Figs. 1A and 1B or Figs. 2A and 2B of the accompanying drawings.

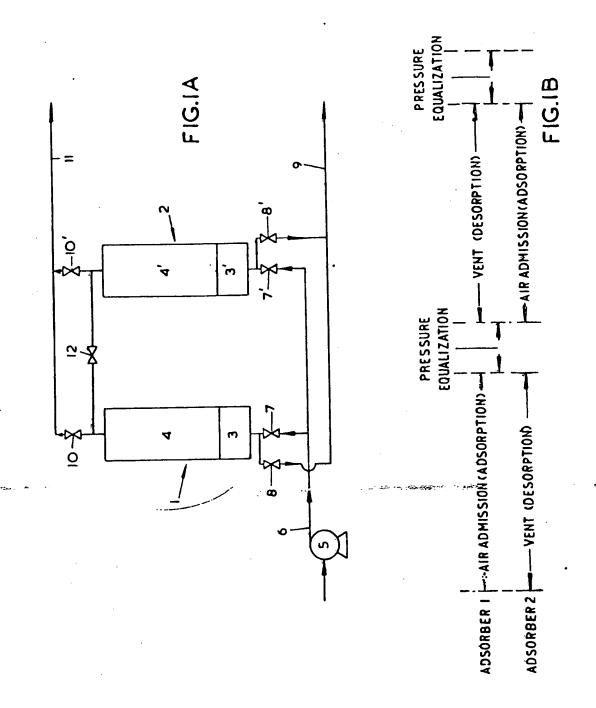
130 11. Apparatus for use in the separation of

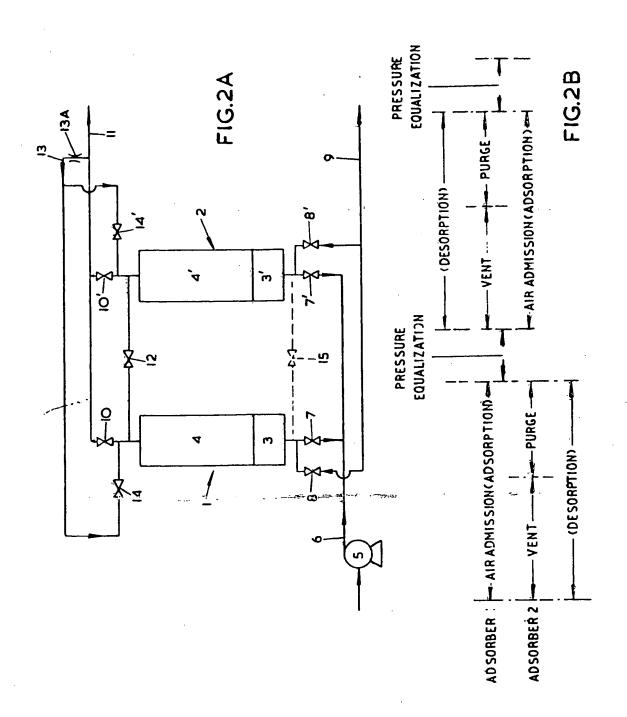












Gas separation

Patent Number:

GB2042365

Publication date:

1980-09-24

Inventor(s):

Applicant(s):

BOC LTD

Requested Patent:

□ GB2042365

Application

GB19790007721 19790305

Priority Number(s):

GB19790007721 19790305

IPC Classification:

B01D53/04

EC Classification:

B01D53/047, C01B21/04D4D

Equivalents:

□ DE3007427, □ JP56045724,

Abstract

A pressure swing adsorption (PSA) process for the separation of a component from a gas mixture also containing water vapour, eg the separation of nitrogen from air, employs adsorbers 1 and 2 each having a first stage 3, 3' containing a desiccant adsorbent and a second stage 4, 4' containing an adsorbent which preferentially adsorbs another component of the mixture eg a carbon molecular sieve which adsorbs oxygen more rapidly than nitrogen. Each adsorber is put through a cycle comprising an adsorption step during which the mixture is passed through the adsorber by a compressor 5 and unadsorbed product is withdrawn through a delivery line 11, and a desorption step during which the adsorber is vented to atmosphere through a waste gas line 9. The pressure reached in the adsorber during such venting is the lowest reached during the entire cycle, no vacuum pump being employed. A

valved line 12 allows pressure equalisation between the adsorbers.



Data supplied from the esp@cenet database - 12